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IN THE UNITED STATES PATENT & TRADEMARK OFFICE

IN RE APPLICATION OF:

ATSUSHI SHIOTA ET AL.

: GROUP ART UNIT: 1712

SERIAL NO: 09/770,289

FILED: JANUARY 29, 2001

: EXAMINER: FEELY, M.

FOR: PROCESS FOR PRODUCING
SILICA-BASED FILM, SILICA-
BASED FILM, INSULATING FILM,
AND SEMICONDUCTOR DEVICE

DECLARATION UNDER 37 C.F.R. § 1.131

COMMISSIONER FOR PATENTS
ALEXANDRIA, VA 22313-1450

SIR:

Now come Atsushi SHIOTA and Kouji SUMIYA, who declare and state that:

1. We are the co-inventors of the above-identified application.
2. Prior to June 11, 1999, we reduced to practice the process for producing a silica-based film claimed in the above-identified application.
3. This reduction to practice is supported by the attached Exhibits A-E.
4. Exhibits A-C are from a laboratory notebook recorded by Atsushi SHIOTA.

Exhibit A is the notebook cover showing that the notebook contains records of "EB / Cure Experiment", i.e., experiments using electron beams for curing. Exhibit B is a record of electron beam exposures of "Siloxane" at doses of "3000-5000 $\mu\text{C}/\text{cm}^2$ ". Exhibit C is a

0105 is the sample number assigned to a particular siloxane

5. Exhibit D shows the dielectric constant ("k"), elastic modulus ("E"), universal hardness ("H") and thickness ("t") of siloxane samples cured with electron doses of 10, 50, 100, 500, 1000 and 3000 $\mu\text{C}/\text{cm}^2$. As discussed above, "LDK-5105" is the sample number assigned to a particular siloxane.

6. Exhibits A-D demonstrate electron beam exposure of siloxane films at doses in a range of from 1 to 200 $\mu\text{C}/\text{cm}^2$ results in films having a dielectric constant of 3 or lower.

7. The feature of "silicon carbide bonds represented by Si-C-Si" is inherent in siloxane films electron beam irradiated at a dose in the range of from 1 to 200 $\mu\text{C}/\text{cm}^2$. The Si-C-Si bonds appear in infrared spectra as a peak at 890 cm^{-1} . Specification at page 27, line 26 to page 28, line 2.

8. Exhibit E is an infrared spectrum of a siloxane film that has received an electron dose in the range of from 1 to 200 $\mu\text{C}/\text{cm}^2$.

9. We hereby declare that all statements made herein of our own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code and that such willful false statements may jeopardize the validity of this application or any patent issuing thereon.

10. Further deponents saith not.

Date: 27 Aug. 2003

Atsushi Shiota
Atsushi SHIOTA

Date: 2 Sep. 2003

Kouji Sumiya
Kouji SUMIYA

Attachments: Exhibits A-E

FOR CLEAN ROOM USE

NEW STACLEAN

Note Book

TEL / 03-5711-1111 / E-MAIL / *clean_experience*

JSR 採用

25-49 Charlie Pans

046/ 328

W

Test : low @ 50

7

50

↓

Sample Si 25-50

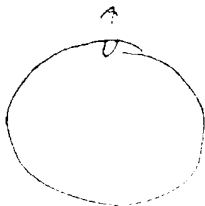
Si/S-N 50

Si/oxide SQR 3,000 = 5,000 $\mu\text{C/cm}^2$

$\phi_1 \sim 6 \text{ min}$

H. L.

N & K



Stage 8.10.10

1. Vacuum
2. Move Stage

Edita Tejnil

3 Task / Baseline Scan

Library / Default. Rile Street

Ling 5-3447.

		Init	6	7	6
UCD-5105/9C1802	Dose	6550 A		1.266	
14SJF1	10	6388.5	40.28	1.260	(0.00000)
16SJG2	80	6006.7	109.74	1.260	(0.00000)
15SJCO	100	5778.2	128.16	1.2612	(0.00062)
11SJ111	500	4840.9	170.06	1.3005	(0.00930)
12SJEO	1,000	4378.5	285.35	1.358	(0.0665)

Energ 5 KeV

Dose 10 μ C/ 50, 100, 1000 μ C/cm⁵⁰⁰

Current 5 mA

Temp. 350°C

Gas Ar

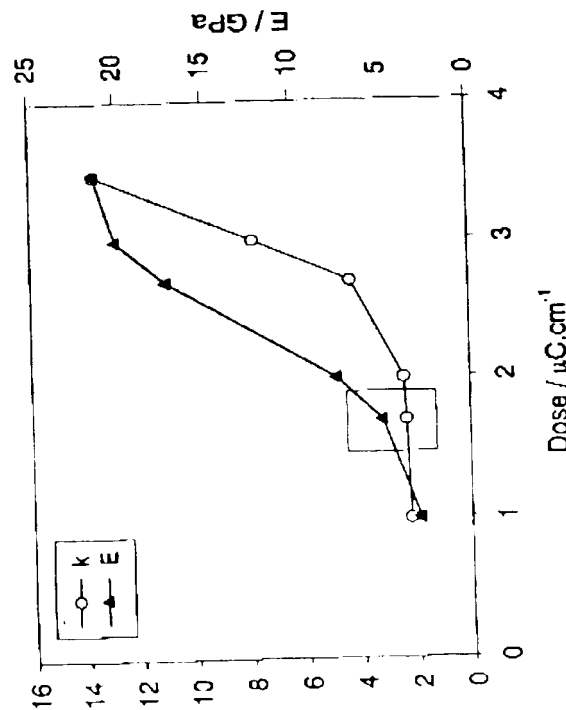
Pressure 10 m Torr

IPE6.676 CMP



Results of Rapid Cure with EB

Rapid Cure by Electron Cure 150



Operation Condition.

Acc. Vol. : 5 keV

Current : 5 mA

Pressure : 10 mTorr

Flow Gas : Ar

Temp. : 400 C

Dose [$\mu\text{C}/\text{cm}^2$]	k	E	H	t [nm]
10	2.31	3.04	0.45	622
50	2.34	5.12	0.77	579
100	2.41	7.52	1.11	542
500	4.29	17.12	1.84	419
1000	7.80	20.01	1.96	394
3000	13.60	21.19	2.15	392
Ctrl	2.27	3.11	0.45	658

(LKD-5105/ JSR Standard Cure Condition)

It was possible to cure rapidly (< 5 min.) using EB.
Optimum dose to increase Young's moduli
without increasing k value was ca. 50 mC/cm².
Moisture adsorption, thermal volatile should be
determined.

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